Retention Time Locked GC-MS Analysis of Phenols

Application

Environmental

Author

Angelica Reese
Agilent Technologies, Inc.
91 Blue Ravine Road
Folsom, CA 95630
USA

Harry Prest
Agilent Technologies, Inc.
1601 California Ave
Palo Alto, CA 94304
USA

Abstract

Comprehensive retention time locking, gas chromatography-mass spectrometry methods for more than 50 phenols and their common internal standards are available. Retention time locking makes the phenol retention times on DB-XLB and DB-5ms columns universal and permanent. "Universal" means that any analyst using an Agilent 6890 Plus Gas Chromatograph and 5973N Mass Selective Detector can reproduce the retention times of every listed phenol. "Permanent" means that the phenol retention times will remain unchanged with typical column maintenance such as column trimming or replacement. This is particularly useful in making definitive assignments of phenol identity from among the many possible isomers that may be present in the samples but may not be on hand in the laboratory standards. A high resolution method and a more rapid "Fast Quant" method were developed for both columns. The high resolution programs have analysis times of 31 and 21 minutes while the "Fast Quant" method runtimes are reduced to 16 and 14 minutes for DB-XLB and DB-5ms columns, respectively. Both columns produce minimal column bleed resulting in improved mass spectral determinations. They also provide excellent separation of the phenols, the DB-XLB column achieving slightly better separation than the DB-5ms.

Introduction

Phenols are widely used compounds and their substituted derivatives are manufactured for use in plastics, drugs, dyes, preservatives, insecticides, fungicides, antiseptics, and disinfectants. They also occur as by-products of various industrial activities such as paper and pulp processing, coal gas liquification, and coke production. This multi-industry use has lead to widespread environmental contamination by phenolic compounds.

Use of chlorine as a bactericide in water treatment results in chlorinated species that rapidly react with phenol to form various substituted chlorophenols. As the degree of substitution increases, acidity, lipophilicity and tendency to bioaccumulate and become toxic to aquatic life increases. These chlorophenols are also added to provide or enhance anti-bacterial properties of various products. Recently, representatives of the German, Danish and Swedish environmental ministries have advised consumers against use of anti-bacterial soaps, many of which contain chlorophenols, in part because of their environmental effects.

Nitrophenols tend to be among the most toxic of the phenols and their alkylated derivatives are commonly used as herbicides and pesticides. It is a startling and disturbing fact that 60 years ago 2,4-dinitrophenol was ingested to cause weight loss. Reputedly, it is becoming popular again despite well documented risks.

As a family, phenolic compounds exhibit a wide range of properties due to the numerous substitution derivatives possible, the most common of which are chlorinated, alkylated or nitro-group derivatives. Consequently, a variety of methods have been developed to test the presence of this important class of compounds in water, soil, sediment, and biota. Recently, a U.S. EPA method (528) has been promulgated that is dedicated to the analysis of 12 common phenols in drinking water. One of the limitations of this method and others is the lack of characterization of other phenol isomers that may be present and undetected or improperly identified.

This work details a gas chromatography-mass spectrometry (GC-MS) approach to the analysis of underivatized phenols. Retention time locking (RTL) is used to make this method universally applicable [1,2]. In other words, any laboratory analyzing samples for phenols can use this method to confidently identify phenolic compounds. This is important because many of the substituted phenols exist as multiple isomers, which exhibit very similar mass spectra and therefore require retention time information for confirmation. This retention time locking (RTL) method allows for the identification of phenols that might otherwise be undetected or possibly misidentified, simply because they are not present in the particular set of standards in use. Obviously, quantification requires standards, but estimating concentrations is possible using the response factors for related phenol isomers via standards on hand.

Experimental

Phenols were obtained from Ultra Scientific (North Kingstown, RI) and AccuStandard (New Haven, CT) as neat compounds and mixtures. Isotopically labeled phenols were acquired from Cambridge Isotope Laboratories (Andover, MA). Dilutions were made in acetone and in dichloromethane (Burdick and Jackson Grade, VWR Scientific).

The configuration and operating parameters of the Agilent 6890 Plus GC ("fast ramping" 220 V option), 7683 Automatic Liquid Sampler, and 5973N MSD with CI option used for acquiring the data are given in Table 1. The relatively recent EPA Method 528 for determination of phenols in drinking water via GC-MS suggests the DB-5ms column [3]. This column is widely used due to its versatility, robustness and low bleed. For similar reasons, the DB-XLB column is also very popular, particularly for the analysis of PCBs and other organochlorine compounds. Method parameters and compound elutions were explored for both columns under the criteria of resolution and total analysis time. This led to the development of two methods for both the DB-XLB and DB-5ms columns. The method details of the GC and MSD programs are given in Table 1.

Phenols are included in a number of U.S. EPA methods including 604, 1625, 1653, 528, 8270, 8041 and others. Each method utilizes different surrogates and internal standards. Method 528 uses 1,2-dimethyl-3-nitrobenzene and 2,3,4,5-tetrachlorophenol as internal standards and 2-chlorophenol-3,4,5,6-d_4, 2,4-dimethylphenol-3,5,6-d_3 and 2,4,6-tribromophenol as recovery surrogates. All these are included in this study. Isotopically labeled phenols, both deuterated and ^{13}C-substituted, provide the most reliable recovery (surrogate) information and have also been included. A variety of internal standards have been applied in the various methods, such as pentafluorophenol, 2,5-dibromotoluene, and 2,2',5,5'-tetrabromobiphenyl. The tetrabromobiphenyl standard requires extending both the GC oven program slightly and the mass scan range beyond the mass range pertinent to the phenols if measuring the molecular ion(s) is desired (as an example, from 50 to 350 *m/z* to 50 to 476 *m/z*). However, the tetrabromobiphenyl method can be used with the "phenol" scan range if one assigns the intense 150 and 310 *m/z* fragments to identify the internal standard. (Technically, if the phenols alone were of interest, the scan range could be confined from 50 to 275 *m/z*, which has the advantage of more scans over the peak.)

The 2,4-dibromophenol was chosen as the locking compound because of its elution in the middle of the oven program, easily distinguished mass spectrum, and low cost.

Table 1. GC Injection, Oven and MSD Parameters for Both Fast Quant and High Resolution Methods Using the DB-XLB and DB-5ms Columns

Injection Parameters

Injection Mode	Pulsed splitless	
Inlet Temperature	200 °C	
Pulse Pressure and Time	25.0 psi	1.00 min
Purge Flow and Time	50.0 mL/min	0.25 min
Gas Saver Flow and Time	20.0 mL/min	3.00 min

DB-XLB Column and Oven Parameters: Fast Quant Method

GC column (P/N: 122-1232)	DB-XLB 30 m × 0.25 mm I.D., 0.25 µm	
Flow and Mode	1.2 mL/min	Constant Flow
Detector and Outlet Pressure	MSD	Vacuum
Oven Temperature Program	40 °C	2.00 min
40 °C/min	100 °C	0.20 min
2 °C/min	110 °C	0.00 min
30 °C/min	340 °C	0.00 min
Oven Equilibrium Time	0.50 min	
Total Program Time	16.37 min	
MSD Transfer Line Temp	320 °C	

DB-XLB Column and Oven Parameters: High Resolution Method

GC column (P/N: 122-1232)	DB-XLB 30 m × 0.25 mm I.D., 0.25 µm	
Flow and Mode	1.2 mL/min	Constant Flow
Detector and Outlet Pressure	MSD	Vacuum
Oven Temperature Program	40 °C	2.00 min
40 °C/min	100 °C	0.50 min
2 °C/min	140 °C	0.00 min
30 °C/min	340 °C	0.00 min
Oven Equilibrium Time	0.50 min	
Total Program Time	30.67 min	
MSD Transfer Line Temp	320 °C	

DB-5ms Column and Oven Parameters: Fast Quant Method

GC column (P/N: 122-5532)	DB-5 ms 30 m × 0.25 mm I.D., 0.25 µm	
Flow and Mode	1.2 mL/min	Constant Flow
Detector and Outlet Pressure	MSD	Vacuum
Oven Temperature Program	40 °C	2.00 min
40 °C/min	100 °C	0.20 min
2 °C/min	105 °C	0.00 min
30 °C/min	340 °C	0.00 min
Oven Equilibrium Time	0.50 min	
Total Program Time	14.03 min	
MSD Transfer Line Temp	320 °C	

DB-5ms Column and Oven Parameters: High Resolution Method

GC column (P/N: 122-5532)	DB-5 ms 30 m × 0.25 mm I.D., 0.25 µm	
Flow and Mode	1.2 mL/min	Constant Flow
Detector and Outlet Pressure	MSD	Vacuum
Oven Temperature Program	40 °C	2.00 min
40 °C/min	100 °C	0.50 min
2 °C/min	120 °C	0.00 min
30 °C/min	340 °C	0.00 min
Oven Equilibrium Time	0.50 min	
Total Program Time	21.33 min	
MSD Transfer Line Temp	320 °C	

Mass Spectrometer Parameters

Tune Parameters	Autotune
Electron Multiplier Voltage	Autotune +400 V
Solvent Delay	4.20 min
Scan Parameters	50 to 340 *m/z*
Quadrupole Temperature	150 °C
Source Temperature	230 °C

Miscellaneous Parts

Septa	5182-0739	BTO septa (400 °C)
Liner	5181-3315	deactivated 4 mm I.D. double taper
GC column ferrule	5181-3323	250 µm Vespel/graphite
MSD interface ferrule	5062-3508	0.4 mm I.D. preconditioned Vespel/graphite

Results and Discussion

Table 2 presents the locked retention times of the phenols using the DB-5ms and DB-XLB columns under the parameters presented in the experimental section. The absolute retention times of the phenols result as a consequence of locking the 2,4-dibromophenol elution time on both columns under all methods. The Fast Quant methods are intended for customers whose analyte list is a subset of the entire phenol list presented here. The High Resolution methods are given for customers with more extensive lists or concerns over possible coelutions or assignments of phenol identity. Both methods are intended for quantitative work.

Method 528 for phenols states that "Any capillary column that provides adequate resolution, capacity, accuracy, and precision can be used. Medium polarity, low bleed columns are recommended ..." Both the DB-XLB and DB-5ms columns meet these criteria. Bleed is extremely low from the DB-XLB

column making it well suited for many GC-MS analyses. The remarks concerning “adequate resolution” are apparently limited to the list of 17 compounds in the method and further qualified by “complete resolution is not necessary ... if unique ions with adequate intensity are available for quantitation.” They do not cite potential failures among the many possible isomeric phenols that lack unique ions. Coelutions are difficult to avoid due to the many possible phenol isomers.

Table 2. Phenol names, CAS numbers, retention times and identifying ions under the acquisition methods are presented in Table 1. All compound absolute retention times are determined by locking the 2,4-dibromophenol retention time to the specified time under each method. On DB-XLB, dibromophenol is locked at 11.320 and 16.220 minutes in the "Fast Quant" and High Resolution methods, respectively. On DB-5ms, dibromophenol is locked at 8.950 and 13.850 minutes in the "Fast Quant" and High Resolution methods, respectively. Tetrabromobiphenyl is listed twice to emphasize the use of different identifying ions appropriate to different scanning ranges; entry #70 is for the scan range 50 to 340 *m/z*.

			Retention Times				
			DB-XLB Fast Quant	DB-5ms Fast Quant	DB-XLB High Resolution	DB-5ms High Resolution	Identifying Ions
#	Compound Name	CAS #	(min)	(min)	(min)	(min)	(*m/z*)
1	2-fluorophenol	367-12-4	4.345	4.135	4.370	4.125	112; 64; 92; 63
2	pentafluorophenol	771-61-9	5.205	4.940	5.360	4.940	183.9; 135.9; 116.9
3	d_5-phenol		5.315	5.070	5.455	5.085	99; 71
4	phenol	108-95-2	5.350	5.085	5.490	5.100	94; 66; 65
5	2-chlorophenol-3,4,5,6-d_4		5.590	5.270	5.730	5.285	132; 133.9; 68; 96
6	2-chlorophenol	95-57-8	5.620	5.300	5.775	5.315	127.9; 129.9; 64
7	2-methylphenol (o-cresol)	95-48-7	6.405	6.025	6.605	6.050	108; 107; 79; 77
8	4-methylphenol (p-cresol)	106-44-5	6.775	6.320	7.005	6.370	107; 108; 77; 79
9	3-methylphenol (m-cresol)	108-39-4	6.815	6.345	7.045	6.375	108; 107; 79; 77
10	2-chloro-5-methylphenol	615-74-7	7.425	6.715	7.690	6.870	141.9; 107; 143.9; 77
11	2,6-dimethylphenol	576-26-1	.630	6.840	7.905	7.035	122; 107; 121; 77
12	2-ethylphenol (o-ethylphenol)	90-00-6	7.980	7.120	8.265	7.550	122; 107; 77
13	2,4-dimethylphenol-3,5,6-d_3		8.400	7.240	8.720	7.770	125; 124; 110; 109
14	2,4-dimethylphenol	105-67-9	8.425	7.255	8.740	7.795	122; 107; 121; 77
15	2,5-dimethylphenol	95-87-4	8.520	7.275	8.850	7.830	122; 107; 121; 77
16	2-nitrophenol-d_4		8.500	7.135	8.825	7.545	143; 69; 85; 113
17	2-nitrophenol	88-75-5	8.560	7.155	8.885	7.585	138.9; 64.95; 81.1; 108.9
18	4-ethylphenol (p-ethylphenol)	123-07-9	8.750	7.425	9.080	8.170	122; 107; 77
19	3-ethylphenol (m-ethylphenol)	620-17-7	8.825	7.440	9.170	8.205	122; 107; 77
20	3,5-dimethylphenol	108-68-9	9.030	7.445	9.410	8.270	122; 107; 77; 121
21	2,3-dimethylphenol	526-75-0	9.085	7.550	9.505	8.455	122; 107; 77; 121
22	2,4-dichlorophenol-d_3		9.060	7.505	9.475	8.305	164.9; 166.9; 66; 101
23	2,4-dichlorophenol	120-83-2	9.110	7.530	9.545	8.350	161.9; 163.9; 97.9; 63
24	2,5-dichlorophenol	583-78-8	9.155	7.550	9.610	8.410	161.9; 163.9; 63; 98.9
25	2,3-dichlorophenol	576-24-9	9.190	7.600	9.660	8.530	161.9; 163.9; 125.9; 63
26	2-isopropylphenol	88-69-7	9.340	7.740	9.900	8.980	136; 121; 103; 91
27	3-chlorophenol	108-43-0	9.360	7.710	9.935	8.910	127.9; 129.9; 65; 99.9
28	4-chlorophenol	106-48-9	9.400	7.715	10.020	8.915	127.9; 129.9; 65; 99.9
29	3,4-dimethylphenol	95-65-8	9.440	7.700	10.100	8.865	122; 107; 121; 77

Table 2. Continued

#	Compound Name	CAS #	Retention Times DB-XLB Fast Quant (min)	DB-5ms Fast Quant (min)	DB-XLB High Resolution (min)	DB-5ms High Resolution (min)	Identifying Ions (*m/z*)
30	1,2-dimethyl-3-nitrobenzene	83-41-0	9.490	7.675	10.175	7.675	151; 134; 77; 106
31	2,6-dichlorophenol	87-65-0	9.700	7.850	10.585	9.215	161.9; 163.9; 63; 125.9
32	2-n-propylphenol	644-35-9	9.720	7.940	10.645	9.585	136; 107; 77
33	2,4,6-trimethylphenol	527-60-6	9.765	7.845	10.750	9.275	136; 121; 135; 91
34	4-chloro-2-methylphenol	1570-64-5	10.470	8.345	12.730	11.105	141.9; 107; 77; 143.9
35	2,3,5-trimethylphenol	697-82-5	10.535	8.375	12.980	11.215	136; 121; 91; 135
36	4-tertbutylphenol	98-54-4	10.615	8.515	13.300	11.905	150; 135; 107
37	4-chloro-3-methylphenol-d_2	59-50-7	10.670	8.480	13.500	11.745	143.9; 109; 143.9; 79
38	4-chloro-3-methylphenol		10.670	8.480	13.500	11.740	141.9; 107; 77; 143.9
39	2,5-dibromotoluene	615-59-8	10.990	8.680	14.640	12.410	249.7; 251.8; 168.8; 170.9
40	2,3,5-trichlorophenol	933-78-8	11.180	8.845	15.605	13.340	195.8; 197.8; 159.8; 199.8
41	2,4-dibromophenol (lock compound)	615-58-7	11.320	8.950	16.220	13.850	251.7; 253.7; 249.7; 63
42	2,4,6-trichlorophenol-d_2		11.390	8.960	16.760	14.075	197.9; 199.9; 201.8; 133.9
43	2,4,6-trichlorophenol	88-06-2	11.405	8.975	16.825	14.120	195.8; 197.8; 131.9; 199.8
44	2,4,5-trichlorophenol-d_2		11.390	8.990	16.760	14.200	97.9; 199.9; 201.8; 133.9
45	2,4,5-trichlorophenol	95-95-4	11.405	9.005	16.825	14.250	195.8; 197.8; 131.9; 199.8
46	2,3,5,6-tetramethylphenol	527-35-5	11.400	9.000	16.950	14.375	150; 135; 91; 151
47	2,3,4-trichlorophenol	15950-66-0	11.465	9.075	17.105	14.510	195.8; 197.8; 159.8; 199.8
48	3,5-dichlorophenol	591-35-5	11.495	9.135	17.775	15.040	161.9; 163.9; 98.9; 63
49	2,3,6-trichlorophenol	933-75-5	11.625	9.180	18.125	14.900	195.8; 197.8; 159.8; 199.8
50	3,4-dichlorophenol	95-77-2	11.700	9.280	19.005	15.410	161.9; 163.9; 98.9; 63
51	3-nitrophenol	554-84-7	12.140	9.600	22.565	16.150	138.9; 65; 93; 81
52	1-naphthol	90-15-3	12.390	9.850	24.175	16.520	143.9; 114.9; 116; 89
53	4-nitrophenol-d_4		12.450	9.865	24.920	16.625	143; 113; 69
54	4-nitrophenol	93951-79-2	12.470	9.875	24.960	16.640	138.9; 65; 109; 81
55	2,5-dinitrophenol	329-71-5	12.500	9.605	24.995	16.045	183.9; 63; 53
56	2,3,4,5-tetrachlorophenol	4901-51-3	12.655	10.040	25.320	16.790	231.8; 229.8; 233.8; 130.9
57	2,3,5,6-tetrachlorophenol	935-95-5	12.655	10.025	25.370	16.775	231.8; 229.8; 233.8; 130.9
58	2,3,4,6-tetrachlorophenol	58-90-2	12.710	10.065	25.550	16.845	231.8; 229.8; 233.8; 130.9
59	2,4-dinitrophenol-d_3		12.710	9.800	25.745	16.420	186.9; 156.9; 110; 54
60	2,4-dinitrophenol	51-28-5	12.730	9.810	25.785	16.445	183.9; 153.9; 106.9; 91
61	3,4,5-trichlorophenol	609-19-8	12.910	10.315	26.285	17.305	195.8; 197.8; 199.8; 132.9
62	2,4,6-tribromophenol	118-79-6	13.155	10.485	26.650	17.450	329.7; 331.7; 327.6
63	2-methyl-4,6-dinitrophenol-d_2		13.280	10.305	27.045	17.235	199.9; 123; 107; 170
64	2-methyl-4,6-dinitrophenol	534-52-1	13.290	10.310	27.055	17.245	197.9; 120.9; 104.95; 167.9
65	pentachlorophenol-$^{13}C_6$		13.660	10.930	27.595	18.045	271.8; 273.8; 269.8; 169.8
66	Pentachlorophenol	87-86-5	13.665	10.930	27.595	18.045	265.7; 267.7; 263.7; 164.8
67	Dinoseb	88-85-7	13.920	11.090	28.065	18.265	210.9; 239.95; 162.95; 146.9
68	2-cyclohexyl-4,6-dinitrophenol	131-89-5	15.620	12.380	29.625	19.660	230.95; 266; 184.95; 192.9
69	2,2',5,5'-tetrabromobiphenyl	59080-37-4	15.625	12.875	29.995	20.165	469.6; 388.7; 471.6; 390.7
70	2,2',5,5'-tetrabromobiphenyl (2)	59080-37-4	15.625	12.875	29.995	20.165	149.9; 309.8; 311.8; 307.8

The DB-XLB method results

Using the DB-XLB column, a single coelution is apparently unavoidable for reasonable oven programs. The 2,4,6-trichlorophenol and 2,4,5-trichlorophenol isomers completely coelute on the DB-XLB under both the Fast Quant and High Resolution methods. Closely eluting peaks in the DB-XLB High Resolution method are the meta and para cresols (≈ 40% resolved), the 2,5-dichlorophenol, 2,4-dichlorophenol and 2,3-dichlorophenol (≈ 50% resolved) the 2,3,4,5-tetrachlorophenol and 2,3,5,6-tetrachlorophenol (> 50% resolved). The DB-XLB Fast Quant Method oven program sacrifices resolution of the 2,3,4,5-tetrachlorophenol and 2,3,5,6-tetrachlorophenol. Additional close elutions occur between 3-chlorophenol and 4-chlorophenol, 3,4-dimethylphenol and 2,3-dimethylphenol, and 2-n-propylphenol and 2,4,6-trimethylphenol.

The DB-5ms method results

Using the DB-5ms High Resolution method, complete coelutions are apparently unavoidable for the 3-methylphenol and 4-methylphenol (meta- and para-cresol), and 3-chlorophenol and 4-chlorophenol for reasonable oven programs. Only partial resolution of 3-ethylphenol, 4-ethylphenol and 3,5-dimethylphenol, 2,4-dimethylphenol and 2,5-dimethylphenol, 2,4-dichlorophenol and 2,5-dichlorophenol, and 2,3,4,5-tetrachlorophenol and 2,3,5,6-tetrachlorophenol can be obtained. This last coelution is particularly troublesome in view of the critical role of the 2,3,4,5-tetrachlorophenol as an internal standard in Method 528. Shortening the oven program in the DB-XLB Fast Quant Method tightens the window between a few compounds such as 2,4,5-trichlorophenol and 2,3,4-trichlorophenol.

General Considerations

Regardless of which column and oven method are used, there are overlapping ions that can affect quantitation ratios, therefore, the user must exercise caution. Table 2 also suggests ions for each of the phenols, although the exact ratios and choices depend on the choice of tuning criteria. This table shows that some confirming ion ratios must be affected by the presence of closely eluting phenols. For example, using the deuterated trichlorophenols as surrogates produces a 198 *m/z* fragment, which is common to the native trichlorophenols. There are similar concerns for other components such as the native and deuterated dichlorophenols.

Two noteworthy deviations from recommendations in Method 528 exist in these methods. The oven programs begin at 40 °C as opposed to the recommended 35 °C. The method states "...GC conditions may be modified, if all performance criteria...are met." Raising the initial temperature to 40 °C avoids the difficulties associated with trying to reach an oven equilibrium temperature of 35 °C when ambient laboratory temperatures are high and so greatly reduces the oven cycle time. This temperature of 40 °C was also demonstrated to work well with standards in dichloromethane as a concession to the continued use of dichloromethane solvent in methods like Method 528 (despite the mandate to reduce the use of chlorinated solvents).

Method 528 also suggests a constant head pressure of 12 to 15 psi. By using constant flow at 1.2 mL/min, the peak shape of the later eluting peaks is dramatically improved, resulting in increased sensitivity and reduced overall runtime. Method 528 contains a peak tailing factor (PTF) performance criteria that requires the acidic and poorly behaving phenols, the 2,4-dinitrophenol, 4-nitrophenol, pentachlorophenol, and 2-methyl-2,4-dinitrophenol, to demonstrate a tailing factor of less than 5 at a concentration of 5 to 10 µg/mL. Figures 1 through 4 show these factors for two of the difficult compounds, the 2,4-dinitrophenol and pentachlorophenol, using the Fast Quant methods for the DB-XLB and DB-5ms columns. Excellent PTFs are achieved for these compounds under these methods. PTFs were less than 1.5 which is much smaller than the required PTF of 5.

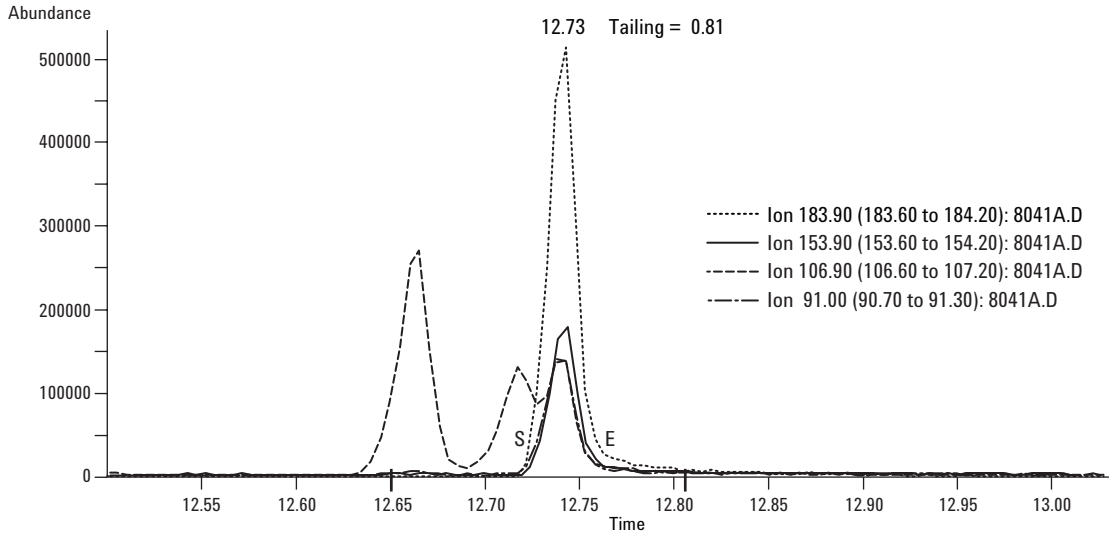

Figure 1. Peak tailing factor 2,4-dinitrophenol at 5 ng/µL on the DB-XLB column with the Fast Quant Method. The PTF is 0.81 (much smaller than the required 5) on a column that had over 100 injections.

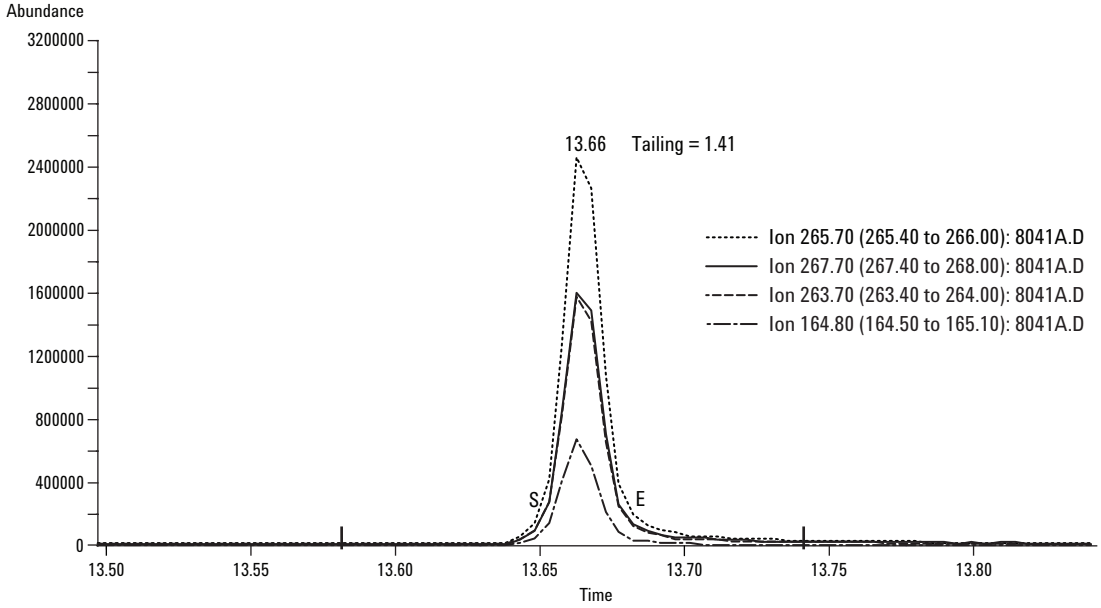

Figure 2. Peak tailing factor for pentachlorophenol at 5 ng/µL on the DB-XLB column with the Fast Quant Method. The PTF is 1.41 (much smaller than the required 5) on a column that had over 100 injections.

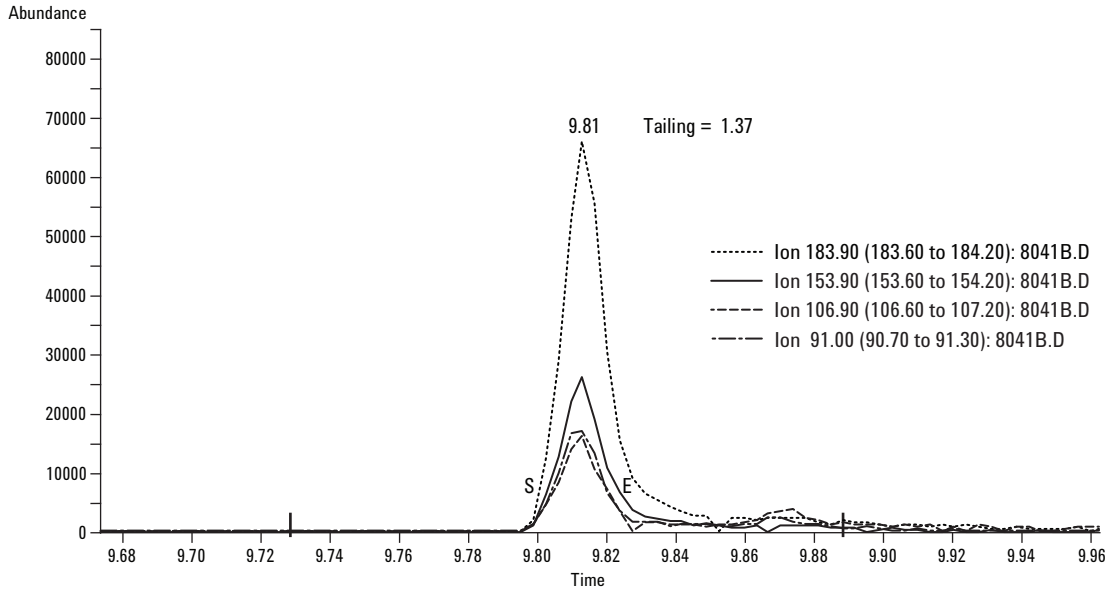

Figure 3. Peak tailing factor for 2,4-dinitrophenol at 5 ng/µL on the DB-5ms column with the Fast Quant Method. The PTF is 1.37 (much smaller than the required 5) on a column that had over 100 injections.

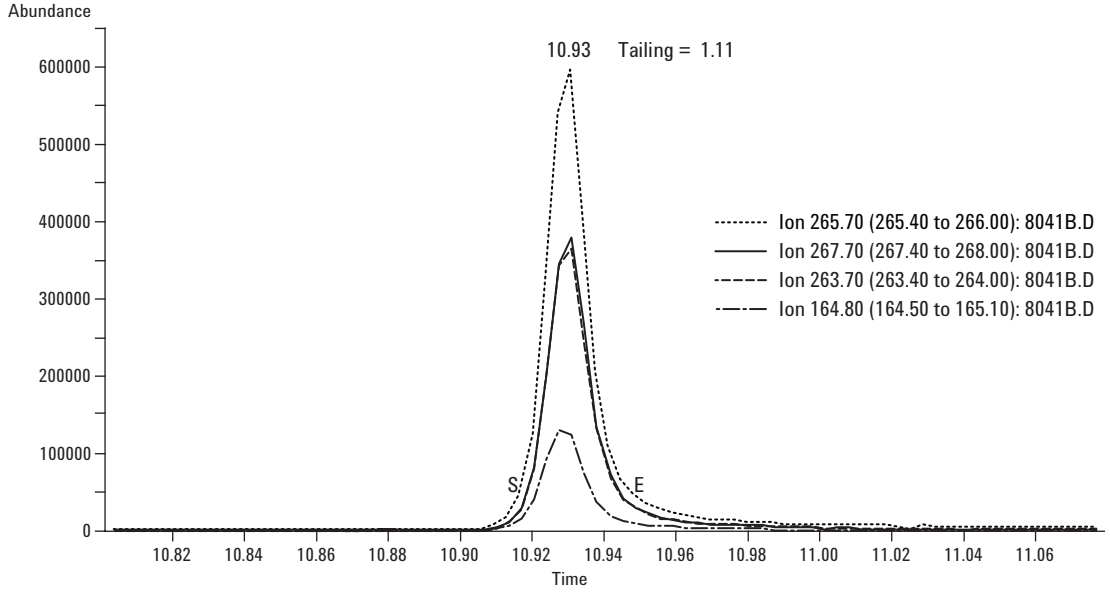

Figure 4. Peak tailing factor for pentachlorophenol at 5 ng/µL on the DB-5ms column with the Fast Quant Method. The PTF is 1.11 (much smaller than the required 5) on a column that had over 100 injections.

"Active" Compounds

The phenols containing nitro-group substituents are well-known to be “active” or difficult compounds that are easily degraded or “lost” in the GC inlet, column, mass spectrometer or GC to MS connection. If the customer wishes to improve the response for these compounds, Agilent supplies liners that directly connect to the GC capillary column and improves the response of the nitrophenols and related poorly performing compounds. These single and double taper direct connect liners (part numbers G1544-80730 and G1544-80700, respectively) have shown a large increase in 2,4-dinitrophenol response over the standard single taper liners. At the minimum, a double taper liner should be used for the phenols. Improvements in response and peak shape are also possible through pressure or flow programming at injection.

Similarly, the new “Ultra” source (part of applications kit G2860A) for the Agilent 5973 and 5973N MSD shows improvement in response and peak shape for the active phenols, especially the dinitro-series. The discussions in the recent Agilent 8270 application note are very pertinent to the analysis of phenols or other active compounds [4].

Conclusions

These methods are completely adaptable to any of the numerous methods requiring phenol quantification. All the phenols and their internal standards used in the U.S. EPA methods, including the recent 528 Method, are listed. A number of phenols not found in any method are also included to offer analysts an opportunity for more complete characterization of phenols and to avoid misidentification. It is apparent that without this precaution there may be mistakes in phenol identifications which will be misleading in environmental studies and limit the usefulness of the data in toxicity assessments. The Fast Quant methods developed on both the DB-XLB and DB-5ms columns are particularly useful when surveying and quantitating a limited number of compounds. The significantly reduced runtimes of 16 minutes (DB-XLB) and 14 minutes (DB-5ms) are quite attractive for rapid, target compound analysis. Obviously, the longer, high-resolution methods provide the best separation. In general, better resolution for the phenols is achieved with the DB-XLB column as compared to the DB-5ms column. Both columns exhibit excellent low bleed characteristics, inertness and robustness, which makes them well-suited to these and related analyses.

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